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ON THE DETERMINATION OF AVAILABLE PHOSPHORIC ACID IN FERTILIZERS CONTAINING COTTON SEED MEAL.

BY F. B. DANCY, A. B.

The term *available* phosphoric acid is used to denote the difference between the *total* phosphoric acid in a fertilizer and the *insoluble*. The *total* phosphoric acid is the entire amount of phosphoric acid, of whatever kind, that the fertilizer contains. The *insoluble* phosphoric acid is, as generally accepted, that phosphoric acid which is left after two grams of the fertilizer, ground to pass a sieve of approximately twenty meshes to the linear inch, have had the *soluble* phosphoric acid extracted with cold water and then been digested for thirty minutes, with agitation every five minutes, at 65° C., with one hundred cubic centimeters of a strictly neutral solution of ammonium citrate of a specific gravity of 1·09, immediately after which digestion they have been thoroughly washed with cold water.

The *available*, then, being the difference between the *total* and the *insoluble*, it follows that *insolubles* being equal, the *available* varies exactly and directly as the *total;* and *totals* being equal, the *available* varies exactly, though inversely, as the *insoluble*.

The *total* is a definite and fixed quantity, and there should, therefore, be no material variation in its determination between the work of accurate analysts. Not so with the others. The

soluble, *insoluble*, *reverted*, and *available* are not fixed and definite quantities. They are dependent on so many conditions of time, temperature, degree of fineness, quality and quantity of solvent, agitation, etc., that it is no matter of wonder that even skillful manipulators vary in their determination. They are the results of methods, and will vary according to the method or the manner of executing the details of the method. But, as has been remarked and as its name implies, the *total* is *all* the phosphoric acid in the material under examination, of whatever kind and in whatever shape or form. It is not what is gotten by a method, but *what there is there*, and any method, therefore, that fails to reach any part or kind of it is not a method for *total* phosphoric acid. It is sometimes said that discrimination is impracticable, and that all fertilizers should be treated equally and alike. The position is untenable. It might as well be urged that in order not to discriminate the plain soda-lime method for the determination of nitrogen should be used on all fertilizers, those containing nitrates as well as those containing organic nitrogen alone, when every one knows that it is inadequate in the presence of nitrates. So a *total* method which is adequate for some kinds of fertilizers, but not for others, cannot be applied to all on the above ground or any other ground, without manifest injustice to those fertilizers for which the method is inadequate.

The Association of Official Agricultural Chemists in their official methods (Bulletin 24, United States Department of Agriculture) give three alternate methods of determining *total* phosphoric acid. There is no distinction made between them, no indication that either is better adapted than the other for any particular class of fertilizers. The presumption is, that they are given as interchangeable and equally allowable for all classes of fertilizers, at the pleasure or option of the operator. It is the purpose of this article to show that one of them, at least, is entirely inadequate for fertilizers that contain cotton seed meal, and that any chemist who uses this method on such fertilizers is in almost certain danger of doing these fertilizers a great injustice.

The method referred to is the second of the three given, namely: "Solution in thirty c. c. of concentrated nitric acid with a small quantity of hydrochloric acid." The writer has not extended his investigation, except imperfectly, to the other two methods. It is only with this one, as applied to cotton seed fertilizers, that this article has to do.

Cotton seed fertilizers are comparatively unknown in the North. It seems, therefore, that the Southern members of the A. O. A. C. must not have been very wide-awake to the interests of a class of fertilizer manufacturers peculiar to their own section of country when they failed to have attached to this method, at the time when it was adopted by their Association as one of three alternate methods, the limitation "not applicable to fertilizers containing cotton seed meal."

Cotton seed meal is readily and entirely soluble in either "nitric acid with a small quanity of hydrochloric acid" or in nitric acid alone. *But such a solution does not give up its phosphoric acid to molybdic solution.* It would appear that certain nitro-organic compounds are formed which prevent the phosphoric acid in the solution from being yielded up to the molybdic precipitant. Whether this is effected by in some way rendering the menstruum a solvent for the phospho-molybdate of ammonia that ought to be formed, or, by holding the phosphoric acid in check, serves thus to prevent such a combination, is not clear. But the fact remains. The attention of the writer was first forcibly directed to it when a sample of cotton seed meal was submitted to him for a determination of the *available* phosphoric acid it contained. A nitric acid solution of two grams of it was made (using also a little hydrochloric acid), the solution being perfect, and a total phosphoric acid percentage of 0.51 found. A duplicate made in the same way yielded 0.54 per cent. Being convinced that there was much more phosphoric acid in the meal than this, and recalling that a short time previously a gentleman had remarked that a friend of his had found materially more phosphoric acid in the ash of cotton seed meal than by acid solution, two grams of the meal were ignited to perfect ash, the ash

dissolved in acid, and a percentage of 3.24 of *total* phosphoric acid found; a duplicate in the same manner yielded 3.20, though in this case the incineration was not quite so perfect, a little char being left. The true per cent. of *total* phosphoric acid in the meal then was 3.24. A solution of two grams made by hydrochloric acid with chlorate of potash also failed by something more than half of getting the full amount. This yielded 1.45 per cent. Next two grams of the meal were taken, washed with cold water in exactly the same manner as when extracting the *soluble* phosphoric acid from an ordinary fertilizer, then digested with citrate solution and again washed exactly as is done in the determination of *insoluble* phosphoric acid. The residue was ignited and the phosphoric acid determined. It was found to be 0.24 per cent. So not only did a cotton seed meal which showed only half a per cent. of *total* phosphoric acid to a nitric acid solution contain in reality three and a quarter per cent., but three per cent. of this three and a quarter per cent. was *available* by the methods of analysis.

A cotton seed meal fertilizer may easily contain one-third cotton seed meal, and, if the meal had the composition of that examined above, would owe one per cent. of its *available* phosphoric acid to the meal. If, therefore, such a fertilizer were to be analyzed by the nitric acid method, it would theoretically show a shortage of nearly one per cent. of *available* phosphoric acid (0.90 exactly). While none of the experiments herein given exhibit as great a disparity as this, some approximate it, and it is believed that a disparity fully equal to this is quite possible. Whether the entire disparity is always due to the retention of the phosphoric acid of the meal alone, or whether in some cases the meal, while holding some of the phosphoric acid of the phosphate in check, gives up more of its own, or whether, in other cases, the phosphoric acid of the phosphate in precipitating carries more of the meal's phosphoric acid down with it than would otherwise go and thus lessens the disparity, is not clear. I am inclined to think that new and fresh meal will exhibit this peculiarity in a greater degree than old meal, though of that I

cannot speak definitely. However that may be, there seems to be not much doubt that the disparity will be greater or less according to various conditions not well understood, and may vary from a third or a half of a per cent. to even as much as one per cent.

Six fertilizers containing cotton seed meal were chosen. They were not laboratory mixtures, but *bona fide* commercial fertilizers on sale in North Carolina. They will be distinguished as 56, 76, 77, 78, 110 and 57. In each the phosphoric acid was determined by dissolving two grams in nitric acid plus a little hydrochloric acid with protracted heating. This is designated in the table below as "acid solution." Then in each the phosphoric acid was determined by incinerating two grams and dissolving the ash in nitric acid. This is designated in the table as "incineration." Following are the results:

Total Phos. Acid....	56	76	77	78	110	57
Incineration.....	10.04	9.77	9.16	9.93	10.07	9.97
Acid Solution.....	9.49	9.13	8.66	9.25	9.56	9.95
Difference.....	.55	.64	.50	.68	.51	.02

No. 57 was an old cotton seed meal fertilizer that had been carried over from last season. The meal had completely changed color to a dark brown, so that to the eye the fertilizer would never have been judged to be a cotton seed fertilizer. In this one alone was there no difference in the results of the two methods.

It is believed that the disparity in all these cases is probably not so great as it should be; that is to say, that the incineration method as here used probably does not give the full content of phosphoric acid, for this reason. The inferiority of the solvent power of nitric acid for phosphates to that of hydrochloric acid is well recognized. I believe that this inferiority is greater when the phosphate has been ignited. To test this an acid phosphate

was chosen, of which two grams were dissolved in nitric acid and hydrochloric acid, and the total phosphoric acid found to be 14.56 per cent. Another two grams were then ignited for about the same length of time that it requires to incinerate two grams of a cotton seed meal fertilizer to complete ash, and then dissolved in nitric acid. The determinations were parallel, so that each received the same amount of heating with acid, which was protracted. In the case of the ignited phosphate, undissolved portions were plainly manifest to the eye, and the percentage found was only 13.62. The undissolved portions were filtered out and readily yielded to hydrochloric acid, giving fulsome precipitate of phosphoric acid. Next, another two grams were ignited and dissolved in hydrochloric acid, when the full content of phosphoric acid was readily yielded. Hence, it is concluded that had hydrochloric acid been used to dissolve the six incinerated fertilizers employed in the above experiments, higher percentages would in all probability have been found. Fusion would have furnished an absolutely certain means of arriving at the undoubted maximum content of phosphoric acid, but time was limited and simple incineration was resorted to on account of expedition; and solution in nitric acid instead of hydrochloric acid was employed after incineration because, as the results were to be compared, it was advisable to employ the same solvent power in each case.

In addition to the six experiments described above attention is called to the following: Two samples of cotton seed fertilizers were procured in which another chemist had made determinations of *total* phosphoric by the nitric acid method. His *totals* were 9.13 and 9.33. I found by incineration and solution in hydrochloric acid 9.85 and 10.13, a difference of 0.72 per cent. and 0.80 per cent. respectively. Taking his own determinations of *insoluble*, the *available* by his analysis was 0.72 per cent. short in the first instance (7.40 per cent. when it should have been 8.12 per cent.), and 0.80 per cent. short in the second instance (7.50 when it should have been 8.30 per cent.). The disparity here is excessive, but the fault is not due to the chemist but to the

method. Being one of the alternate methods of the A. O. A. C., he had no reason to doubt its adequacy.

Having looked at one side of the determination of *available* phosphoric acid in fertilizers containing cotton seed meal, let us turn to the other, namely, the determination of *insoluble*. As almost the entire content of phosphoric acid in the meal has been shown to be available, it might be anticipated that there would be likely not to be much difference in the determination of *insoluble* whether the citrate-extracted residue is first incinerated or dissolved directly in acid. Such was found by experimentation to be the fact. In fact, in every case but one (and this was the same fertilizer that was the exception to the *total* rule, namely, the old one brought from the previous season) a slightly higher *insoluble* was found by dissolving in acid directly than after incineration.

The details of the two methods of determining the *insoluble* were these: After filtering from the citrate and thoroughly washing, the filter and contents were in the first instance incinerated and the ash dissolved in nitric acid (designated in the table below as "incineration"), and in the second instance the filter and contents were introduced directly into flasks and completely dissolved with nitric acid and a little hydrochloric acid (the "acid solution" of the table below). The samples used were the same as those used in the total experiments, with the exception of 110, which was not used.

Insoluble Phosphoric Acid...	56	76	77	78	57
Incineration	0.84	1.80	0.86	0.87	1.59
Acid Solution.....	1.00	1.91	1.28	1.05	1.42
Difference.....	.16	.11	·42	.18	.....

Whether the excess of *insoluble* by direct acid solution was due to mechanical loss in the incineration by the other method (which in these instances is not at all apprehended, though it is believed that care is necessary to with certainty guard against

such loss), or whether, as is much more likely, it was due to imperfect solution of the ignited phosphate by nitric acid (a danger already pointed out), is not positively shown. But it should be remarked that in the case of 77, which is the case of greatest disparity, the undissolved portions were manifest to the eye, and it is the writer's conviction that had hydrochloric acid been used instead of nitric, there would have been no material difference in the results of the two methods, either in these particular determinations, or in any other determinations. It is regarded as certain, therefore, that there will be found no material difference in the *insoluble* by which ever way determined. This being true, the *available* will vary directly and exactly as the *total;* and, therefore, by just so much as a *total* is short on account of the inadequacy of the nitric acid method when used on cotton seed meal fertilizers, by just so much will the *available* be short.

Now a few words as to what method *is* adequate and a very few experimental determinations on this point, and the subject will for the present be laid aside. It is a matter of regret to the writer that the time was not at his disposal for more extended experimentation on the subject. Nevertheless he is none the less convinced, on account of the limited number of experiments here presented, of the truth of the point urged.

In order to compare the *total* as made by several different methods sample 110 was chosen. This sample was sent by a fertilizer company, who took a fair sample of a large lot of goods at their factory, thoroughly mixed the sample and sent one-half to the writer and one-half to a chemist in another State. The *total* was determined first by fusing two grams of the fertilizer with a mixture of equal parts of carbonate of soda and nitrate of potash, as giving with certainty the maximum content, and furnishing a standard for comparison. It was then determined by incineration and solution in hydrochloric acid, by incineration and solution in nitric acid, by solution direct in hydrochloric acid with chlorate potash, and lastly, by solution direct in nitric acid and a little hydrochloric acid. Following are the results:

Fusion, ...	10.14 per cent.
Incineration and solution in HCl., . . .	10.09 "
" " " HNO_3, . .	10.07 "
Solution direct in HCl. + $KClO_3$, . . .	10.11 "
" " HNO_3 + little HCl., .	9.56 "

From the above it seems that in this case all the methods save the nitric acid method were adequate, but it is not apprehended that this will hold good in all cases. For, while here incineration and solution in nitric acid sufficed, there is certainly the danger of imperfect solution already referred to; and while here also solution in hydrochloric acid with potassium chlorate sufficed, it has already been shown that this method failed to extract, from the meal alone, more than half its content of phosphoric acid. Fusion is, of course, always adequate, but too time-consuming, if any other adequate method less so is at hand. It seems probable that incineration and solution in hydrochloric acid furnishes all that could be desired.

It is in point to add that in this case the writer reported to the manufacturers a *total* of 10.11 per cent., this being an average of the first four determinations. The *insoluble* found by incineration and solution in hydrochloric acid was 1.47, making an *available* of 8.64. It is not known what method the chemist used to which the other half of this sample was sent, but his *total* was 9.72, his *insoluble* 1.50 and his *available*, therefore, 8.22. Note that the two *insolubles* are practically the same, and that, therefore, his *available* is less than the writer's by practically the same amount that the writer's *total* is greater than his.

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THE DISTRIBUTION OF BORACIC ACID AMONG PLANTS.

BY J. S. CALLISON.

Attention was first drawn to the possibility of boracic acid occurring as a normal constituent in certain plants by its discovery in normal California wine.

Baumert* first drew attention to this in 1885, and his results were confirmed by Rising† and Crampton.‡

These observations were extended to other vines by Baumert,§ Soltsein‖ and Ripper.¶

It was shown by Baumert and Ripper, especially, to be almost invariably present in wines of all countries, in the stalks, and even in the wild vine (Soltsein). As Crampton says, "There can no longer be any question, therefore, in view of this mass of evidence, that boracic acid is a normal and frequently occurring constituent of the grape plant." He then asks the question, if boracic is so universal a constituent of the grape plant, why not of other plants as well? In answering this question he examined certain plants, as the peach, water-melon, apple, sugar-beet and sugar-cane. Thinking this distribution of boracic acid to be a question of decided importance, I have extended the examination, to many other ashes than those mentioned by Crampton, coming from other classes of plants than fruits, though including also some of the fruits.

The results of my work have confirmed the conclusions that boracic acid is very widely distributed in the vegetable kingdom,

*Landw. Versuchstat. **33,** 39-88.
†Report of Sixth Viticultural Convention, 1888.
‡Amer. Chem. Journal, Vol. II, 227.
§Zeitschrift fur Naturwissenchaften, 1887, and Ber. d. deutsch. Chem. Ges. **21,** 3290.
‖Pharmaceutische Zeitung **33,** No. 42, p. 312.
¶Weinbau und Weinhandel: Organ des deutschen Weinbauvereins, No. 36, 1888.

and that there seems to be a power of selection on the part of the plants, some having no affinity for and not taking up the boracic acid, though growing on the same soil from which other plants absorb it. We are forced to the belief that it is contained in the soil and that the plants draw it from that source, yet in no case could I detect its presence in the soil, and in no well water examined could I get a test for it. It is easily possible that other waters might contain it. It would be of great interest to extend this examination of natural waters so as to see whether the boracic acid in the soil is in a soluble form or not. It may be present in a form soluble in plant solvents and insoluble in natural waters. Of course it is possible, also, that it is present in such great dilution that the amount of water evaporated failed to give the qualitative tests. My examination of different fertilizing materials revealed the fact that several of the commonest contained boracic acid in appreciable amounts, and hence, by this means, it is being constantly added to soil under cultivation.

Of course stable manure, coming from grain and straw, restores to the soil the boracic acid contained in these.

A further important fact brought out by this research is that every sample of caustic alkali examined contained boracic acid. These samples were from the best known manufacturers, and were marked chemically pure. As these alkalies, especially potassium hydroxide, are used in most of the methods for determining boracic acid, it is manifestly of great importance to secure them free from it as an impurity. I think it possible that the boracic acid in these samples, or at least in some of them, may have come from the glaze of vessels used in their manufacture or from the bottles containing them.

The method of testing was that used by Meisell for the detection of boracic acid in milk.* About one gram of the ash was treated with strong hydrochloric acid and boiled a few minutes to insure solution. It was then filtered from the unburnt carbon, insoluble silica, etc. The filtrate was evaporated to dryness

*Konig's Nahrungsmittel, **2**, 250.

on a sand-bath and the heat continued until the excess of acid was driven off and the residue assumed a white appearance. This was then moistened with very dilute hydrochloric acid (1 : 100), a few drops of tincture of turmeric were added, and the entire mass dried down on a water-bath. The appearance of the cherry-red or cinnabar-red color was taken as an evidence of boracic acid. The residue was then used for the flame tests. This was made by placing a part of it upon a strip of platinum, moistening with alcohol and igniting. The green flame flashes, best observed by blowing out and relighting the alcohol, were regarded as confirmatory of the turmeric tests. The flame test is, however, not so delicate as the latter, as has also been observed by Crampton.

The relative and absolute delicacy of these tests was also approximately determined. The flame reaction was still clear with .01 gm. of boracic acid, but could not be gotten with .001 gm. This latter amount gave the color reaction, but .0001 gm. failed to give it.

These figures apply, of course, only to boracic acid treated under like circumstances to the ash. 10 cc. of a solution of boracic acid of known strength was evaporated in a small porcelain dish and the residue manipulated exactly as in Meisell's test.

The porcelain dishes used in this research, and other apparatus where the presence of boracic acid might be suspected, were carefully tested and shown free from anything that could conflict with the tests.

The appended table gives the substances examined and the results of the tests. In many cases the tests were carefully repeated to insure accuracy. The specimens were chosen so as to represent as many different classes of plants as possible. In the case of caustic alkalies the evaporation and testing were done in platinum vessels.

The experiments with various chemicals, pure and commercial, were begun with the idea of seeing whether those which might have come from some plant source, contained this substance so generally present in the plants themselves. The caustic

alkalies were examined with a view to their use in the quantitative determination of boracic acid. Some other chemicals, as ferric oxide, were examined to see if they would in any way interfere with the test when present.

The table which follows will need no further explanation. I wish to express my obligations to Dr. F. P. Venable for his guidance and supervision of my work in this research.

Fruits.

No.	Common Name.	Botanical Name.	Flame Reaction.	Color Reaction.
1	Fig, green	Ficus Carica	Yes (faint)	Yes
2	leaves		No	No
3	branches		Yes	Yes
4	Persimmon, green	Diospyros Virginiana	Yes (faint)	Yes
5	leaves		No	No
6	Pear, branches	Pyrus Communis	Yes (faint)	Yes
7	leaves		Yes	Yes
8	Apple, pulp	Malus	Yes (faint)	Yes
	seed		Yes (faint)	Yes (faint)
9	Peach, branches	Prunus Communis	Yes (faint)	Yes
10	leaves		Yes	Yes
11	Honey Locust, pods	Gleditschia triacanthos	Yes	Yes
12	Lemon, pulp	Citrus Limonum	No	Yes
13	rind		Yes	Yes
14	Orange, pulp	Citrus Aurantium	Yes	Yes
15	rind		Yes (faint)	Yes
16	seed		No	Yes
17	Banana	Musa Sapientum	No	Yes
18	Dates, dried		Yes (faint)	Yes (faint)
19	seed		Yes (faint)	Yes (faint)
20	Cherry, branches	Prunus Ceracus	No	No
21	leaves		Yes (very faint)	Yes (faint)
22	Cocoanut, shell	Coco Nucifera	No	Yes (faint)
23	Raspberry, leaves	Rubus Strigosus	No	Yes
24	Blackberry, stalk	Rubus Villosus	No	Yes
25	leaves		No	Yes
26	Strawberry, leaves	Fragaria Vesca	Yes	Yes
27	Grape, Concord leaves	Vitis Labrusca	Yes	Yes
28	stalk		Yes	Yes
29	Wild, leaves	Vitis Cordifolia	No	Yes
30	stalk		No	Yes

Vegetables and Grains.

31	Pea	Pisum Arvense	Yes	Yes
32	Beet, root	Beta Vulgaris	Yes	Yes
33	leaves		Yes	Yes
34	Salsify, root	Fragopogon Porrifolium	Yes (very faint)	Yes (faint)
35	leaves		Yes	Yes
36	Celery	Apium Graveolens	Yes	Yes
37	Wheat, grain	Triticum Vulgare	Yes (faint)	Yes
38	green stalk		Yes	Yes
39	Oats, grain	Avena Sativa	No	Yes (faint)
40	green stalk		Yes	Yes
41	Corn, grain	Zea Mays	No	No
42	cob		No	Yes (very faint)
43	stalk		No	No
44	blades		No	No
45	Maple, branches	Acer Rubrum	Yes	Yes
46	leaves		Yes	Yes
47	Willow, branches	Salix Nigra	Yes	Yes
48	Mulberry, wood	Morus rubra	Yes	Yes (faint)
49	bark		No	No
50	Walnut, branches	Juglans Nigra	Yes	Yes
51	Sumach, berries	Rhus copallina	Yes	Yes
52	branches		Yes	Yes
53	Pine, long leaf, branches	Pinus Australis	Yes	Yes
54	leaves		Yes (faint)	Yes
55	short leaf branches	Pinus Mitis	Yes	Yes
56	leaves		Yes	Yes
57	Dogwood, branches	Cornus Florida	Yes (faint)	Yes
58	Cedar, branches	Juniperus Virginiana	Yes	Yes
59	leaves		Yes	Yes
60	Oak, branches	Quercus alba	Yes (faint)	Yes
61	leaves		Yes	Yes
62	acorn		Yes (faint)	Yes
63	Sycamore, branches	Plantanus Occidentalia	Yes	Yes
64	Elm, branches	Ulmus	Yes	Yes
65	Black How, branches	Viburnum Prunifolium	Yes	Yes
66	fruit		Yes	Yes

67	Hickory, branches	Carpa	Yes	Yes
68	nut		Yes (faint)	Yes (faint)
69	Magnolia, branches	Magnolia Grandiflora	Yes	Yes
70	leaves		Yes	Yes
71	Holly, branches	Ilex Opaca	Yes	Yes
72	leaves		Yes	Yes
73	berries		Yes	Yes
74	Paulonia, buds	Paulonia imperialis	Yes	Yes
75	pods		Yes	Yes
76	Osage Orange	Maclura Aurautiaca	Yes (faint)	Yes

Miscellaneous Plants.

77	Tobacco, stalk	Nicotiana Tabacum	Yes	Yes
78	leaves		Yes	Yes
79	Cotton, lint	Gossypium herbaceum	Yes (very faint)	Yes (very faint)
80	seed		Yes (very faint)	Yes
81	stalk		Yes	Yes
82	Azalea Indica, leaves	Azalea Indica	No	Yes (faint)
83	root		No	No
84	Chrysanthemum, stalk	Chrysanthemum Pompone	No	Yes (faint)
85	flower		No	Yes (faint)
123	Rose, branches		Yes	Yes
86	Bamboo	Smilax Rotundifolium	Yes	Yes
87	Sorrel	Oxalis Stricta	Yes	Yes
88	Lucerne	Medicago Sativa	Yes	Yes
89	Clover, red	Trifolium Pratense	Yes	Yes
90	white	Trifolium Repens	Yes	Yes
91	Orchard Grass	Dactylis Glomerata	Yes (faint)	Yes
92	Blue Grass	Poa Compressa	Yes	Yes

Soils, Waters and Fertilizers.

No.	Common Name.	Remarks.	Flame Reaction.	Color Reaction.
93	Alluvial	2 kilos taken	No	No
94	Sandy, rich	" "	No	No
95	Sandy, poor	" "	No	No
96	Red Clay		No	No
97	Well Water, No. 1	48 liters taken	No	No
98	No. 2	56 " "	No	No

99	Water from small stream	36 litres taken	No	No
100	Bone and Peruvian Guano		Yes	Yes
101	Peruvian Guano		Yes (faint)	Yes
102	Bone Ash		No	No
103	Sodium Nitrate	Crude	No	No
104	Muriate of Potash	From Stassfurt	Yes	Yes
124	Kainite	For Fertilizers	No	Yes
125	Kelp		Yes	Yes

Chemicals.

105	Potassium Nitrate	C. P.	No	No
106	Potassium Carbonate	C. P.	No	No
107	Potassium Chlorate	C. P.	No	No
108	Potassium Carbonate	Commercial	Yes	Yes
109	Potassium Phosphate	C. P.	No	No
110	Sodium Nitrate	C. P.	No	No
111	Sodium Carbonate	C. P.	No	No
112	Calcium Carbonate	C. P.	No	No
113	Ferric Oxide		No	No
114	Concentrated Lye	Commercial	No	Yes (faint)
115	Potassium Carbonate	Pearlash	No	No
116	Potassium Hydroxide	C. P., Trommsdorf	Yes	Yes
117	" "	by alcohol, Marquart	No	Yes
118	" "	by baryta, Schuchardt	No	Yes
119	" "	" Eimer & Amend	No	Yes
120	" "	C. P., free from Al_2O_3, SiO_2 and SO_3, Eimer & Amend	No	Yes
121	Sodium Hydroxide	C. P.	Very faint	Yes
122	" "		No	Yes (very faint)
126	Ammonium "		No	No

University of North Carolina,
May, 1890.

ON THE OCCURRENCE OF BORACIC ACID AS AN IMPURITY IN CAUSTIC ALKALIES.

BY F. P. VENABLE AND J. S. CALLISON.

In the course of a research upon the distribution of boracic acid in the ashes of plants, it was decided to make some quantitative estimations of the boracic acid present. The reagents to be used were first themselves tested for boracic acid, and, much to our surprise, no sample of the caustic alkalies could be procured free from it. Specimens coming from some of the most noted manufacturers, Schuchardt, Marquart, and Trommsdorff, purified by alcohol or by baryta, were found to contain boracic acid, and sometimes in decidedly appreciable amounts. No quantitative determination has been made, but, judging from the known delicacy of the qualitative tests, the amount must have often exceeded 0.1 per cent., and was probably much greater.

As the caustic alkalies, especially potassium hydroxide, are frequently used in the methods for the quantitative determinations of boracic acid,* this presence of it as an impurity may be a serious source of error. The knowledge of it is important on other grounds as well.

*See, for instance, Morse & Burton, Amer. Chem. Jour. X, 154. Ztschr. and Chem. **25,** 202.

UNIVERSITY OF NORTH CAROLINA,
May, 1890.

THE DETERMINATION OF CRUDE FIBER.

BY W. A. WITHERS.

This was begun to ascertain, if possible, some of the causes of the discrepancy in results obtained by different chemists for crude fiber. The samples were Timothy hay and cotton seed meal. They were not extracted with ether previous to treatment with alkali and acid, as is customary in ordinary analyses. Except when so stated, the solutions used were 1¼ per cent. H_2SO_4, and 1¼ per cent. Na_2O solutions, the time of boiling 30 minutes, and the results in per cent. calculated on the dry sample.

The following questions were considered:

1. Amount of Na_2O neutralized.

By titration before and after treatment, an average of 5 different determinations gave an equivalent of .114 grams Na_2O neutralized by Timothy hay for every gram of substance taken, showing that only a very small portion of the Na_2O added enters into chemical combination, and part of this is in the saponification of the fat which would usually be extracted with ether.

2. Sulphuric acid neutralized.

Residues from Timothy hay, after treatment with Na^2O solution, were taken. It was found that for each gram of substance originally taken, .01 gram of H_2SO_4 was neutralized, showing that the H_2SO_4 does not enter into chemical combination at all.

3. Effect of different strengths of Na_2O solution.

This was tested on Timothy hay with the following results:

Time of treatment	45 minutes.		1½ hours.	
Na_2O (grams) used per gram of substance	2.34	1.27	1.27	1.00
Residues	33.67	35.26	33.34	35.06
	33.28	35 36	33.70	35.30
	...	...	33 88	...
Average	33.48	35.31	33.64	35.18
Difference due to different strengths of Na_2O solution	1.83		1.54	

This shows that the stronger the solution, or the more of the same solution used, the smaller is the per cent. of fiber.

4. Effect of time in treatment with Na_2O solution.

In the filtration a simple asbestos filter was used. After boiling the substance for 30 minutes with the alkali, the residue was detached from the filter as completely as possible, and subjected to another similar treatment with a new portion of the reagent. This method avoids very largely errors of analysis that would be shown had different samples been taken and treated for different lengths of time. Duplicate determinations A and B were made:

Treatments with Alkali.	Timothy Hay.				Cotton Seed Meal.			
	A.		B.		A.		B.	
	Res.	Dis.	Res.	Dis.	Res.	Dis.	Res.	Dis.
1	36 96	63.04	36.21	63.79	4.46	95 54	4.66	95.34
2	33.49	3.47	32.79	3.42	3.95	.51	4.14	.52
3	31.85	1.64	31.17	1.62	3.26	.69	3.47	.67
4	30.53	1.22	29.82	1 35	2.99	.27	3.09	.38
5	29.74	.79	29.03	.79	With H_2SO_4.			
6	28.78	.96	28.34	.69	2.39	.60	2.41	.68

These treatments represent about 50 minutes contact with the alkali, being about 10 minutes to raise to boiling, 30 minutes boiling, 5 minutes standing, and 5 minutes filtering. The first treatment required from 10 to 15 minutes in filtering, thus making the contact longer. In the case of Timothy hay, therefore, this difference is about .07 per cent. of fiber per minute, and of cotton seed meal .01 per cent.

5. Effect of time in treatment with H_2SO_4 solution.

The residue after treatment with alkali was treated in the same way as above. Timothy hay was used.

From Na_2O treatment with	34.70	65.30	33.92	66.08	28.78	71.22	28.34	71.66
acid ....1.	29.10	5.60	28.33	5.59	25.59	3.19	25.38	2.96
2.	28.20	.90	27.60	.73	24.79	.80	24.73	.65
3.	27.81	.39	27.25	.35	23.91	.88	24.08	.65
4.	27.32	.49	26.78	.47				

The time of contact with acid was about 40 minutes, which makes a difference of nearly .02 per cent. of fiber for every minute of treatment with acid.

Ladd (5th N. Y. Ex. Sta. Rep.) has shown that the degree of heat employed causes a difference in results. The above work shows that differences are also due to amount and to the concentration of the different reagents used, and to the time of contact. To secure agreement in results, uniformity in all these conditions must be attained.

Granting this can be done with different workers, the question of accuracy of results confronts us. Without going into a review of the work of different chemists bearing on this point, I should like to call attention to the above tables under 4 and 5. One of two conclusions must be drawn from this work, viz.: 1st, That if half an hour's treatment with each alkali and acid is sufficient to give the per cent. of crude fiber, then crude fiber is soluble in both alkali and acid, and that to a somewhat considerable extent; or, 2d, that if crude fiber is not soluble in these reagents, then 6 treatments with alkali, and 3 subsequent treatments with acid, are not sufficient to separate it. From either of these conclusions it is evident that the method lacks accuracy, and is at best only a rapid method of rough approximation to the truth.

6. Ash in the residue.

Six samples were taken for this:

2 treated with alkali 45 minutes and acid 1 hour.
2 " " " 1½ hours " " ½ "
2 " " " " " " 1 "

In no case was any ash found in the residue.

7. Nitrogen in the residue.

The albuminoid equivalent of this was found to vary from 4 per cent. to 5 per cent. with one treatment with alkali. The acid subsequent treatment did not remove any more, but subsequent treatments with alkali left no nitrogen.

CHEMICAL LABORATORY,
CORNELL UNIVERSITY.

SOME MODIFICATIONS OF THE METHOD FOR DETERMINING CRUDE FIBER.

BY W. A. WITHERS.

With the method now in use for determining crude fiber (*vide* Bul. 19, 1888, U. S. Dept. of Ag.) there is little trouble in the manipulation, except in cases of feeding-stuffs, in which the percentage of albuminoids is rather large. In all such cases, however, the albuminoids precipitated by the treatment with H_2SO_4 seriously impede the filtration, rendering it very slow, and from this longer contact causes a part of the crude fiber to be dissolved.

This difficulty can be obviated by treating the substance with the Na_2O solution before rather than after the treatment with H_2SO_4 solution.

To test this modification, I compared it with the ordinary method, on samples of Timothy hay and cotton seed meal. In both cases clear filtrates were secured by the modification in from 15 to 30 minutes, while with the ordinary method the filtration with the hay required about the same time; but the cotton seed meal from 18 to 24 hours, and then the filtrate was not clear. With Timothy hay the two methods gave practically the same results. With cotton seed meal the ordinary method gave 2.68 per cent. and 2.91 per cent., an average of 2.80 per cent., while the modification gave, with five determinations, 3.63 per cent., 3.58 per cent., 3.69 per cent., 3.49 per cent.,

3.44 per cent., an average of 3.57 per cent. The determinations in hay show the modification gives reliable results. In the case of cotton seed meal, however, we see that 77 per cent. crude fiber is dissolved by the ordinary method, on account of the longer contact produced by the slow filtration, being more than 20 per cent. of the whole amount.

For accuracy and, therefore, agreement of results, as well as time-saving, this modification seems to recommend itself.

Rapid Filter.—The asbestos filter gives good results if covered with a layer of glass wool. Care should be taken, however, not to add too much liquid at a time, as this may cause the glass wool to become displaced. The coarse asbestos gives the best results.

Ladd (N. Y. Ex. Sta. Rep., 1887, p. 378) finds that when the asbestos filter was returned with the residue a larger per cent. of fiber was found, and the two reasons given are that it prevents so thorough contact with the reagents, and that it necessitated lowering of the temperature to prevent bumping. When, however, the substance is first treated with alkali there is no tendency to bump in the acid treatment, and consequently no need of lowering the temperature. As to how much the first mentioned cause will affect the results, I have not tested.

Prevention of Frothing in Boiling.—This can be done by directing a moderate blast of air into the flask through a small glass tube (1.5 mm. diam.). The constant level of the liquid can be maintained by adding boiling water from time to time, or by a reflux condenser.

CHEMICAL LABORATORY,
CORNELL UNIVERSITY.

LINVILLE METEORITE.

[From the American Journal of Science, Vol. XXXVI, October, 1888.]

ART XXIX.

ON THREE NEW MASSES OF METEORIC IRON.

BY GEORGE F. KUNZ.

I. Meteoric Iron from Linville Mountain, Burke County, North Carolina.

A mass of meteoric iron* was found on Linville Mountain, Burke county, N. C. (long. 81° 35′ W. of Greenwich, lat. 35° 40′ N.), about the year 1882. It was handed to a country blacksmith in the vicinity, who sold it to a tourist miner, and by him it was sold to Mr. Norman Spang, of Etna, Pa., who, not being a collector of meteorites, has kindly allowed it to come into my possession.

This meteorite weighs 428 grams; the original weight was 442 grams (15½ ounces), the remainder having been used for analysis and for etching; it is 2⅗ inches (65 mm.) long, 1⅖ inches (35 mm.) high, and 2½ inches (38 mm.) wide. One side is rather rough, and the other pitted with very shallow pittings. Traces of the black crust of magnetic oxide of iron are still visible, and although the mass is not rusted, yet small drops of chloride of iron have collected in the deep clefts; in one of these was also found a spider's egg-case, suggesting that the iron is either a recent fall, or had been found on the surface of the ground.

In cutting a piece from the lower side, the blacksmith has destroyed a good deal of the surface as well as the crust, on account of the toughness of the iron. The iron admits of a very high polish, yielding a rich nickel color, which, under the glass and by reflected light, shows an apparent net-work of two distinct bodies.

*Exhibited at the New York Academy of Sciences, Dec. 5th, 1887.

When bromine water or diluted nitric acid is applied to a polished surface of the iron, it blackens and does not show the Widmanstätten figures. If this black deposit is washed off, an orientated sheen appears, which resembles that of the Green county iron, described by Blake,* and the iron in the Port Orford, Oregon, meteorite, as figured by Brezina and Cohen in "Die Structur und Zusammensetzung der Meteoreisen, etc."† Almost the entire surface has, under the glass, the appearance of a mesh-work of which the irregularly rounded centers have been eaten out. At a few places on both sides of a crack is a small piece of troilite 3 mm. by $1\frac{1}{2}$ mm., through which are scattered small patches of meteoric iron that after etching exhibit beautiful octahedral markings so delicate as to be invisible to the naked eye, and somewhat like those of the Tazewell, Claiborne county, meteorite, though not more than one-tenth the thickness.

The following analysis was kindly furnished by Mr. J. Edward Whitfield, of the United States Geological Survey, through the courtesy of Prof. F. W. Clarke:

	Linville, Whitfield.	Tazewell,‡ Smith.	Bear Creek,§ Smith.
Iron	84·56	83·02	83·89
Nickel	14·95	14·62	14·06
Cobalt	0·33	0·50	0·83
Copper	0·0	0·06	trace
Sulphur	0·12	0·08	.....
Carbon	trace	.....	.....
Phosphorus	"	0·19	0·21
Magnesium	.....	0·24	.....
Silica	none	0·84	.....
	99·96	99·57	98·12

Dr. F. A. Genth has kindly furnished the following analysis:

Iron	85·83
Cobalt	0·73
Nickel	13·44
	100·00

*Amer. Journal Sci., III, Vol. xxxi, p. 41.
†Stuttgart, 1876, Lieferung I, Tafel VI.
‡Original Researches, 1884, p. 439.
§Amer. Journal Sci., II, Vol. xix, p. 153.

It most closely resembles the Tazewell, Claiborne, and Bear Creek, Col., meteorites in composition. I herewith take pleasure in thanking Mr. Norman Spang for his kindness in allowing me to secure the iron and for the facts of its discovery; also, Mr. J. Edward Whitfield and Prof. F. W. Clarke for the analysis.

II. On the Meteoric Stone from Ferguson, Haywood County, North Carolina.

Mr. W. A. Harrison, of Ferguson, North Carolina, says that about six o'clock, on the evening of July 18, 1889, he noticed a remarkable noise west of him, and that fifteen minutes later he saw something strike the earth, which, on examination, proved to be a meteoric stone, so hot that he could scarcely hold it in his hand five minutes after it fell. Two-thirds of its bulk was buried in the earth when found. This stone was sent to the writer, and was unfortunately lost in New York City during the month of December.

The stone was slightly oblong, covered with a deep, black crust, which had been broken at one end, showing a great chondritic structure with occasional specks of iron. Its weight was about eight ounces, and it very closely resembled the meteoric stone from Mocs, Transylvania. It remained in the writer's possession so short a time that it was not properly investigated; but still the mere mention of a fall, which had been so carefully observed, is thought to be well worthy of publication.

III. Meteoric Iron from Bridgewater, Burke County, North Carolina.

The Bridgewater, Burke county, meteorite was found by a negro plowman, two miles from Bridgewater Station, in the western part of Burke county, near the McDowell county line in North Carolina. Latitude, 35° 41′; longitude, 81° 45′ W. of Greenwich. The negro thought that it must be either gold or silver, and took it to some railroad laborers, who broke it in two pieces, one of which weighed ten-and-a-half, and the other eighteen-and-a-half pounds, together 30 pounds, equal to 13.63

kilos. The iron measures 22.5 x 15 x 10 cm. (9 x 6 x 4 inches).

Traces of black crust very much oxidized are still visible on the surface. The iron is highly octahedral in structure, and the mass was readily broken by the laborers who found it. Between the cleavage plates schreibersite is visible.

On etching a polished surface of this iron with dilute nitric acid, the characteristic Widmanstätten figures were shown. The iron belongs to the caillite group, and resembles those of the Cabin Creek and Glorietta Mountain in structure.

The specific gravity of a fragment was found to be 6.617. The following analysis was kindly furnished by Prof. F. P. Venable, of the University of North Carolina:

Fe	88.90
Ni	9.94
Co	.76
P	.35
Cl	.02
	99.97

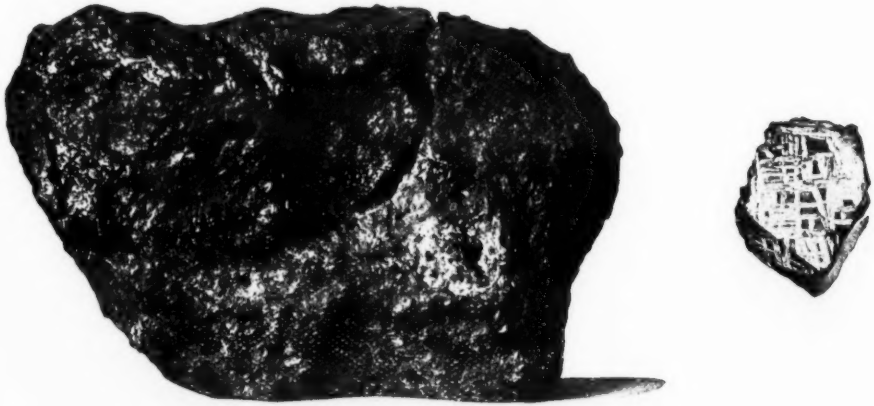
The nickel is the mean of two determinations, 9.74 and 10.14, on different parts of the sample.

The cobalt also of two determinations, .85 and .67.

The iron is the mean of four determinations, some of which were not very closely agreeing, as the crust could not be entirely removed from the samples taken.

The phosphorus and chlorine are single determinations.

The author takes great pleasure in thanking Mr. T. K. Bruner for his courtesy in obtaining the information and the iron for him, and in thanking Professor F. P. Venable for furnishing the analysis.



BRIDGEWATER METEORITE.

TWO NEW METEORIC IRONS.

BY F. P. VENABLE.

I. FROM ROCKINGHAM COUNTY, N. C.

This mass was reported to have fallen about the year 1846, near the old "Mansion House," Deep Springs Farm, in Rockingham county, N. C. One of the old negro servants related to Mr. Lindsay, the present owner of the farm, that "the rock fell on a clear morning, and struck the ground about a hundred yards back of the garden. It frightened every one very much. Colonel Jas. Scales, the proprietor at that time, and Mr. Dillard took a man and went to the spot, dug in about four or five feet and got it out." It lay about the house as a curiosity for several years, when it ceased to be of any more interest, and was thrown aside. After Mr. T. B. Lindsay bought the farm he kept the meteoric mass for several years upon his porch. In the fall of 1889 he presented it to the State Museum. The indentation in the earth, where it is reported to have struck, is still pointed out.

The weight of the mass was 11.5 kilos. It had somewhat the outline of a rhomboid, measuring 270 x 210 mm., and having a thickness varying from 10 to 70 mm. It is coated with oxidation products to a depth, in places, of several millimetres. These give the whole mass a dull, reddish brown color. The surface is irregularly pitted with broad shallow pits. It is somewhat concave on one side. On being polished and etched it gave faintly the Widmanstätten figures. It belongs to the class of sweating meteorites, beads of deliquesced ferric chloride appearing on the surface. This lawrencite, so-called, is evidently unevenly distributed through the mass. Analyses from different portions gave different amounts of chlorine. In one boring it was noticed that the metal near the surface (within $2^{cm.}$) gave a decided percentage of chlorine, while that coming from the deeper part of the drill hole (3—5 cm. from surface) gave no appreciable amount.

The analysis gave:

Fe	87.01
P	.04
SiO_2	.53
Cl	.39
Ni	11.69
Co	.79
	100.45

II. FROM HENRY COUNTY, VA.

This meteoric iron was found by Nathaniel Murphy, in Henry county, Va., about four miles from the Pittsylvania county line, and one-half mile north of the dividing line between North Carolina and Virginia, near to Smith River. Murphy found the stone in a ploughed field in the latter part of the spring of 1889. He gave it to Colonel J. Turner Morehead, of Leaksville, N. C. Together with Colonel Morehead he searched over the farm, but could find nothing similar to this piece. Colonel Morehead sent the mass to Dr. H. B. Battle, of Raleigh, N. C. It weighed 1.7 kilos, and the detached pieces, mainly crust, weighed 0.22 kilos. This crust broke off along certain lines by a sort of cleavage, and the main mass is permeated with cracks, not irregular and zigzag, but as distinct and regular, almost, as if it were a piece of crystallized gypsum. This cleavage is in two directions. The laminae vary in thickness, but many are about $\frac{1}{2}$ mm. The color of the surface is dark bluish black, mixed with much red-rust coming from the lawrencite. Parts of the soil apparently still clung to the mass. It measured 60 x 70 x 75 mm., taking the greatest lengths in the three directions. Here and there scales or spots of bright silvery sheen were to be seen. It contains a good deal of ferric chloride, and is rapidly crumbling. On polishing one of the faces, the Widmanstätten figures (coarse) came out very plainly, no etching being necessary.

The analysis resulted as follows:

Fe	90.54
Cl	.35
SiO_2	.04
P	.13
Co	.94
Ni	7.70
	99.70

UNIVERSITY OF NORTH CAROLINA,
May, 1890.

A LIST AND DESCRIPTION OF THE METEORITES OF NORTH CAROLINA.

BY F. P. VENABLE.

So far as can be learned, twenty-three meteorites have been reported as found in North Carolina. Facts with regard to these have been collected under many disadvantages and with great difficulty. A complete list of references in scientific literature has proved an impossibility; still a great many such references are given. It is also impracticable now to trace all of the possessors of portions of these meteorites. They have been divided often into many pieces, and widely scattered. Only occasional clues to their whereabouts can be gotten at the present time. One fact is made apparent, and that is, that nearly all have passed out of the State, not even fragments being preserved here.

It will be noticed that, with the exception of one from Nash county, all of the reported meteorites have come from Western North Carolina. That many of these came to the light at all has been due to the intelligent energy of General T. L. Clingman, to whom the State owes so much already for bringing to notice her minerals and other possessions.

It has been thought best to include in this list all reported falls and finds. In the case of all proved to be non-meteoric, or about which doubt exists, note is made under the proper heading. If these doubtful ones be eliminated, as well as those not belonging properly to the State, the number is reduced to about twenty. There is doubt, however, whether the number should be as great even as this, as there is cause for thinking the Madison county, and, perhaps, some of the Buncombe county finds may belong to the same fall. Still the number is large when we bear in mind the comparatively small number of recorded meteorites for the whole earth. Huntington in his catalogue (1887) places the number at 424.

I must express my acknowledgments to Mr. S. C. H. Bailey, of New York, for most valuable assistance rendered in this compilation.

1.

ALEXANDER METEORITE.

Locality—Cedar Creek, Alexander county. Not analyzed.

This iron, weighing about fifty-six grams, was given by General T. L. Clingman to Mr. S. C. H. Bailey, of New York, about the year 1875. It has not been analyzed, nor have I been able to learn more of its origin. The piece, Mr. Bailey writes, is evidently a fragment from a larger mass, and is sufficiently characteristic to be distinguishable from any other iron, though it more nearly resembles the Sarepta (Russia) iron.

Literature—

Possessor—Bailey (56 grams).

2.

ASHE METEORITE.

Locality—Ashe county. *Analyst*—Shepard (?)

The only reference that can be found to this mass is the following, coming from the American Journal of Science:

"A fragment of meteoric stone from Ashe county, N. C., examined at the same time, was found to contain a marked quantity of this principle (chlorine), the presence of which, however, was accounted for by the fragment having been in contact with a bag of salt as it was carried home by the person who found it."

It is possible that this is the same as the Grayson county, Virginia, meteorite.

Literature—Am. Jr. Sc., 1st Ser. XLVIII, p. 169; Rep. Am. Met., p. 34; Buchner, p. 168.

Possessor—Unknown.

3.

Asheville Meteorite.

Locality—Asheville, Buncombe county. *Analyst*—Shepard.

This meteorite was presented by Dr. J. F. E. Hardy to Dr. C. U. Shepard for examination. It weighed between nine and ten ounces, and had been detached from a rounded mass nearly as large as a man's head, which mass was found loose in the soil about five or six miles west of Asheville, on the farm of a Mr. Baird, near the south-western base of an elevation of land five hundred feet high. Dr. Hardy was of opinion that other masses existed at the same place.

The specimen had a distinctly crystalline structure, approaching a flattened octahedron. The surface had a dissected or pitted appearance, occasioned by the removal of portions of the external laminæ during its separation from the original mass. The cavities were perfectly geometrical in shape, being rhomboidal, tetrahedral, or in the figure of four-sided pyramids. Sections of the external laminæ loosened broke up easily into regular octahedra and tetrahedra very exact in form. Some of the plates separated into leaves nearly as thin as mica, and delicately sticated in every direction.

The specific gravity of different pieces varied from 6.5 to 7.5, and even as high as 8.

ANALYSIS.

	I.	II.
Iron	96.5	94.5
Nickel	2.6	5.
Silicon	.5	0.3
Chlorine	.2	
Chromium, Sulphur, Cobalt, Arsenic	in traces.	
	99.8	99.8

Analysis I is taken from Am. Jr. Sc., Vol. xxxvi, p. 81. Analysis II is also credited to Shepard, and is taken from Clark's List, p. 55. It seems to be an analysis of the original lump, from which the smaller fragment described above was taken.

Literature—Am. Jr. Sc., 1st xxxvi, p. 81; Clark, p. 55; Rep. Am. M., 1848, p. 24; Buchner, p. 163; Partsch, p. 116; Jahresber, 1847–48, p. 1309; Huntington, p. 60; Smithsonian Report, p. 261; Min. and Min. Loc., p. 14.

Present Possessors—Amherst, 276 grams; Göttingen, 1.50; Shep. Cab. in National Museum, 2.95; London, 114.9; Vienna, 271; Berlin, 13.66; Paris (Nat. Hist.), 72; and in private collections: Gregory, 2; Siemascho (St. Petersburg).

4.

Black Mountain Meteorite.

Locality—Black Mountain, Buncombe county. *Analyst*—Shepard.

The Black Mountain meteorite was found at the head of the Swannanoa River, near the base of Black Mountain, towards the eastern side of Buncombe county. It was given by Dr. Hardy to Colonel Nicholson, of South Carolina. By the latter it was given to Dr. Barratt, of the same State, and from him it was secured by Dr. Shepard. It seems to have been picked up about 1839. The fragment weighed twenty-one ounces, and was evidently a portion of a larger mass. Its texture was highly crystalline, having all the laminæ (which were usually thick) arranged conformably to the octahedral faces of a single individual. There was evidence of the existence of very minute veins of magnetic iron pyrites. The mass contained several rounded and irregular nodules of graphitic matter, with which again were found large pieces of iron pyrites.

Specific gravity, 7.261.

Iron	96.04
Nickel	2.52
Cobalt	traces.
Insoluble matter, sulphur and loss	1.44
	100.00

Literature—Am. Jr. Sc., 2d Ser. iv, p. 82; Rep. Am. M., p. 28; Jahresber, 1847-'48, p. 1310; Buchner, p. 180; Clark, p. 34; Huntington, p. 56; Kerr Appendix, p. 56; Min. and Min. Loc., p. 14.

Present Possessors—Amherst, 243 grams; Yale, 15; London, 71.5; Vienna, 45; Paris (Nat. Hist.), 5; Dorpat, 19; Neville (now Calcutta), 29; and in private collections: Baumhauer, 44; Siemascho.

5.

Hominy Creek Meteorite.

Locality—Hominy Creek, Buncombe county. *Analysts*—Shepard and Clark.

The Hominy Creek meteorite, sometimes referred to simply as Buncombe county meteorite, was secured for Dr. Shepard by Hon. T. L. Clingman. The original discoverer was a Mr. Clarke, and the date of the discovery seems to have been 1845. It was found in a field near the base of Mount Pisgah, some ten miles west of Asheville. Another much larger piece was reported to have been found in the same field. The mass weighed twenty-seven pounds. It was rather flat on one side, while its other sides were irregular, with cavities and various inequalities. Externally, it bore resemblance to a cinder from a blacksmith's fire. It measured eleven inches in length by seven in breadth, and was four in thickness at the thicker end, while at the other extremity it is not above two and a half. On the lower edge it thinned down to about one inch. Its surface was rather jagged than pitted with regular depressions. In color it was various shades of brown to black, and somewhat variegated with an ash-colored earthy matter, derived undoubtedly from having served for a considerable time as a support for fuel in the fire-place of a farmer's kitchen. Upon the under side there adhered over a few inches a crust of an earthy, black amygdaloid nature, scarcely distinguishable, unless freshly broken, from the iron itself, and in one spot a few grains of a dull, yellowish gray olivine were noticed. Etched surfaces, excepting where the structure is highly vascular, exhibit the most delicate Widmanstätten figures. Specific gravity, 7.32.

SHEPARD.	
Iron	98.19
Chromium and Cobalt	traces.
Nickel	0.23
Carbonaceous, insoluble matter and loss	1.58
	100.00

CLARK.	
Fe	93.225
Ni. } Co. }	.236
Ca. } Sn. }	.099
Mn.	?
Si.	.501
Mg	?
P	?
S	.543
Graphite } Schreibersite }	4.765

The yellowish olivine grains consisted of silicic acid, lime, magnesia and ferric oxide.

This is placed among the pseudo-meteorites in the British Museum, and does not seem to be recognized as a meteorite by other authorities.

Literature—Am. Jr. Sci., 2d Ser. IV, p. 79; Rep. Am. M., p. 25; Jahresber, 1847–'48, p. 1310; Buchner, p. 175; Clark, 22; Min. and Min. Loc., p. 14; Kerr Appendix, p. 56.

Present Possessors—Yale, British Museum.

6.

Linville Meteorite.

Locality—Linville Mountain, Burke county. *Analyst*—Whitfield.

A mass of meteoric iron was found on Linville Mountain, Burke county, about the year 1882. It was handed to a country blacksmith in the vicinity, and, passing through several hands, finally came into the possession of Geo. F. Kunz, Esq., of New York.

The original weight was 442 grams. It was $2\frac{3}{5}$ inches long, $1\frac{3}{5}$ inches high, and $2\frac{1}{2}$ inches wide. One side was rather rough, and the other pitted with very shallow pittings. Traces of the black crust of magnetic oxide of iron were still visible. The mass was not rusted, and small drops of chloride of iron were noticed in the deep clefts, and in one of them was found a spider's egg-case, suggesting either that the iron was a recent fall, or that it had been found on the surface of the ground.

On being polished it gave a rich nickel color, and showed an apparent net-work of two distinct bodies. The Widmanstätten figures were not given on etching. The analyses gave:

Iron	84.56
Nickel	14.95
Cobalt	0.33
Sulphur	0.12
Carbon	trace.
Phosphorus	trace.
	99.96

Literature—Am. Jour. Sc., 3d Ser. XXXVI, p. 275.

Present Possessor—Geo. F. Kunz, Esq.

7.

Bridgewater Meteorite.

Locality—Bridgewater, Burke county. *Analyst*—Venable.

This meteorite was described by Kunz. It was found by a negro two miles from Bridgewater Station, in the western part of Burke county, near the McDowell county line. It was broken by some laborers into two pieces, one weighing ten and a half and the other eighteen and a half pounds. The original lump, therefore, weighed thirty pounds or 13.63 kilos. The iron measures 22.5 x 15 x 10 cm.

Traces of black crust, very much oxidized, are still visible on the surface. The iron is highly octahedral in structure. Between the cleavage plates schseibersite is visible. Widmanstätten figures gotten on etching.

ANALYSIS.

Iron	88.90
Nickel	9.94
Cobalt	.76
Phosphorus	.35
Chlorine	.02
	99.97

Literature—Trans. N. Y. Acad. Sci., Jan., 1890; Mitchell Soc., Vol. VII, p. 29.

8.

Cabarrus Meteorite.

Locality—Post Farm, Cabarrus county. *Analyst*—Shepard.

The fall of this meteorite was described by J. H. Gibbon, Esq., of the United States Branch Mint at Charlotte. On October 31, 1849, at 3 P. M., a sudden explosion, followed at short intervals by two other reports, and by a rumbling in the air to the east and south, was heard in Charlotte. Five days later news was brought of the fall of a meteoric mass on the farm of a Mr. Hiram Post in Cabarrus county, some twenty-five miles distant. This stone weighed nineteen and a half pounds, was bluish and gritty in appearance, of irregular form, eight

inches long, six broad and four thick, bearing marks in spots of recent fracture, but otherwise black, as if it had been exposed to heat and smoke, the black color being relieved where the crust had been broken, and a little of the clayey soil in which it was buried in its descent still adhered to it. Lustrous metallic points appeared through the ground color. Mr. Post had heard the explosion and heard the stone strike about three hundred yards off with a dull, heavy jar of the ground. The stone had splintered a pine log lying on the ground. It was buried under some ten inches of soil.

It is further described in the Huntington Catalogue as a "stone—dark gray with light grains and thickly sprinkled with iron. Fragment showing dull black crust" (referring to the fragment in the Harvard collection).

The specific gravity was 3.60—3.66.

Nickeliferous iron (with chrome)..	6.320
Iron sulphide.....	3.807
Silica	56.186
Ferrous oxide.....	18.108
Magnesia	10.406
Alumina.....	1.707
Lime, soda, potash and loss	3.394

Literature—Am. Jour. Sci., 2d Ser., IX, p. 143; X, p. 127; Buchner, p. 79; Kerr Appendix, p. 56; Huntington, p. 69; Smithsonian Rep., p 263; Min. and Min. Loc., p. 16.

Present Possessors—Amherst (mass larger than two fists); Harvard, 168 grams; National Musuem (Shep. Cab.), 343.6; London, 385.5; Vienna, 138; Berlin, 133; Göttingen, 33; Paris, 42; Dorpat, 29; Dresden, 7; Bologna, 3; Yale, 2.31; Calcutta, 52; Gregory, 152; Baumhauer, 51; Siemascho.

9.

Caldwell Meteorite.

Locality—Caldwell county. *Analyst*—Venable.

A small piece of iron was found among the specimens for the State Museum, labeled, in Dr. Kerr's handwriting (roughly, on an old piece of paper), "Meteoric iron from Caldwell county." The reference in his note-book said it was received from a Col. Scilly. It has proved impossible to learn anything further

about it. It was probably set aside by Dr. Kerr for examination, but the coming on of his final illness prevented it. The piece is about the size of a silver dollar, is still fairly bright; has evidently been hammered out thin and weighs five grams. It gave no figures on etching and a qualitative analysis revealed iron as the only metal present. It is probably of terrestrial origin.

Present Possessor—N. C. State Museum.

10.

CASWELL METEORITE.

Locality—Caswell county.

This stone fell at 2 P. M. on 30th January, 1810. It was described by Bishop Madison (of Williamsburg, Virginia) as resembling other meteoric stones, especially the one which fell at Weston, Connecticut, in 1807. It was not only attracted by the magnet, but was itself magnetic.

Whether the stone is still preserved anywhere and who possesses it is as little known as anything further with regard to its characteristics.

Literature—Gilb. Ann., 41, 1812, 449; Chladin, 291; Buchner, 27; Kerr App., 56; Min. and Min. Loc., p. 13.

11.

DAVIDSON METEORITE.

Locality—Lick Creek, Davidson county. *Analysts*—Smith and Mackintosh.

This was found on July 19, 1879, by Mr. Gray W. Harris on his land near Lick Creek, Davidson county. It was somewhat pear-shaped and weighed $2\frac{3}{4}$ pounds. Its outward color is dark brown, not rusty. The original crust was almost entirely hammered off by the finder, but a little remaining showed a peculiar slaty lamellar structure and readily broke into flakes. Some cavities in this crust were lined with mammillary forms, and it had many seams with a vitreous luster. It failed to give the Widmanstätten figures.

The analysis gave:

Iron	93.00
Nickel	5.74
Cobalt	0.52
Phosphorus	0.36
Sulphur	traces.
Chlorine	traces.
Copper	traces.
Carbon	not determined.
	99.62

From four analyses by Smith and Mackintosh. Meteorite in the possession of Hidden.

Literature—Am. Jour. Sci., 3d Ser. xx, p. 324; Min. and Min. Loc., p. 17.

Present Possessors—Main mass in Vienna, 887 grams; London, 20; Paris (Nat. Hist.), 41; Harvard, 6; Bailey, 38; Hidden, ? Nat. Mus. (Shep. Cab.), 9.72.

12.

GUILFORD METEORITE.

Locality—Guilford county. *Analyst*—Shepard.

This was secured by Prof. Olmsted in 1820 from a man who told him that it had been detached from a large mass weighing twenty-eight pounds, which was wrought by a blacksmith of the neighborhood into horse nails. The fragment weighed seven ounces. It was a distinct crystal in the form of an octahedron. The axis measured three inches, the angle at the summit was 60°, that at the base 122°. Its structure was distinctly foliated, the laminæ being uniformly one-twentieth of an inch in thickness and arranged parallel with the planes of the octahedron. The exact locality of the find is not given, but it was stated that it was found some ten or fifteen miles distant from the locality where the Randolph county specimen was found. It exhibited, when etched on tarnished or polished surfaces, very perfect Widmanstätten figures.

Analysis:

Iron	92.750
Nickel	3.145
Iron sulphide	0.750

Literature—Am. Jour. Sci., XVII, p. 140; XL, p. 369; Clark, 61; Rep. Am. M., p. 24; Partsch, p. 114; Huntington, p. 52; Min. and Min. Loc., p. 13; Kerr Appendix, p. 56.

Present Possessors—Amherst, 2.15 grams; Yale, 20; London, 15; Vienna, 8; Göttingen, 8; Calcutta, 10.5.

13.

Haywood Meteorite.

Locality—Haywood county. *Analyst*—Shepard.

This fragment, weighing one-eighth of an ounce, was sent to Dr. Shepard by Hon. T. L. Clingman, accompanied by the following remark: "It was given me by a person in Haywood county whose father had obtained it in that region, but without his being able to designate the locality. It is evidently meteoric iron, but is perhaps from some mass already known."

The fragment was highly crystalline and somewhat tetrahedral in form. One side was polished and etched. It displayed a marked character, and one which has no analogue among meteoric irons. It was irregularly veined by a black ore, which was not acted upon by acids and which presented all the properties of magnetite.

Specific gravity=7.419. It contained iron, sulphur, phosphorus, chromium, and was rich in nickel.

Literature—Am. Jour. Sci., 2d Ser. XVII, p. 327; Min. and Min. Loc., p. 15; Kerr Appendix, p. 56; Buchner, p. 189.

Present Possessor—Amherst, 10 grams.

14.

Haywood Meteorite.

Locality—Ferguson, Haywood county.

Mr. Harrison, of Ferguson, N. C., noticed about 6 P. M., July 18, 1889, a remarkable noise west of him. Fifteen minutes later he saw something strike the earth, and this on examination proved to be a meteoric stone, so hot that he could scarcely hold it in his hand five minutes after it fell. Two-thirds of its bulk was buried in the earth when found. The

stone was slightly oblong, covered with a deep black crust which had been broken at one end, showing a great chondritic structure with occasional specks of iron. Its weight was about eight ounces and it very closely resembled the meteoric stone from Mocs, Transylvania. It was unfortunately lost in New York before examination.

Literature—Mitchell Soc., Vol. VII, p. 29.

15, 16, 17.

Madison Meteorites.

Locality—Duel Hill, Madison county. *Analysts*—Smith and Burton.

There are several meteoric masses attributed to Duel Hill and to Jewel Hill, Madison county. The similarity of these names in pronunciation, and apparent confusion between them, led to inquiry as to their exact location. The result of the inquiry is that at present no Jewel Hill is known in this county. There was a Jewel Hill, at one time the county-seat, but its name was changed to Duel Hill and the county-seat removed to Marshall. These two are therefore one and the same locality.

Several masses have been found there.

No. 16. Found in 1856 and recorded as preserved in the Amherst collection. It weighed forty pounds. No analysis has been found. Amherst has two pieces—one of 600 grams and one of 167½ grams.

No. 17. This meteorite was presented to Dr. Smith in the year 1854 by Hon. T. L. Clingman. It came from Jewel Hill, Madison county, of that State. There was a great deal of thick rust on the surface, with constant deliquescence from chloride of iron. Its form and surface indicated that it was entire. Its dimensions were 7 by 6 by 3 inches, with a number of indentations. Its weight was eight pounds thirteen ounces. The analysis gave:

Iron	91.12
Nickel	7.82
Cobalt	.43
Phosphorus	.08
Copper	trace.
	99.45

Literature—Scient. Res., p. 317, 410; Min. and Min. Loc., p. 15; Kerr App., p. 56; Huntington, p.

Present Possessors—London, 130.2 grams; Vienna, 4; Paris (Nat. Hist.), 104; Göttingen, 38; Dorpat, 17; Harvard, 160; Yale, 5.610; Nat. Mus., 91; Nat. Mus. (Shep. Cab.,) 31.85; Calcutta, 16; Bailey, 11.4; Gregory, 40.

18.

Locality—Duel Hill, Madison county. *Analyst*—Burton.

This mass was found in August, 1873, on the land of Robert Farnesworth, near Duel Hill, Madison county. It was lying on a hill-side where it had been used in supporting a corner of a rail fence, which was quite decayed at the time of finding. It is said to have weighed, when first found, about twenty-five pounds. Two or three pounds were hammered off as specimens before it fell in the hands of Prof. Burton, who analyzed it.

Mr. Farnesworth reported that a similar mass weighing about forty pounds had been found about a mile farther west, probably about 1857, and had since disappeared. Efforts to find it again were unsuccessful.

This meteorite was of a rounded irregular shape, $9 \times 6\frac{1}{2} \times 3\frac{1}{2}$ inches, and weighed twenty-one pounds. On being etched, it gave the usual markings, though indistinctly. Distinct particles of schreibersite were irregularly disseminated over the surface. Deliquescent beads of lawrencite were also to be seen.

Specific gravity=7.46.

Iron	94.24
Nickel	5.17
Cobalt	0.37
Phosphorus	0.14
Copper	trace.
Residue	0.15
	100.07

The residue contained SiO_2, Fe., Cr., Ni. and P.

Literature—Amer. Jour. Sci., 3d Ser. XII, p. 439; Min. and Min. Loc., p. 15.

Present Possessors—London, 12 grams; Vienna, 160; Harvard, 222; Baumhauer, 29; Bailey, 3; Gregory, 1.

19.

Nash Meteorite.

Locality—Castalia, Nash county. *Analyst*—Smith.

This meteorite fell May 14, 1874, at 2:30 P. M, near Castalia (lat. 36° 11′, long. 77° 50′). Its fall was accompanied by successive explosions and rumbling noises, lasting about four minutes. The stones that fell must have exceeded a dozen or more—three only were found and they gave evidence that the territory over which the fragments fell was ten miles long by over three miles wide. Although occurring in the day, the body appeared luminous to some observers. The three stones found weighed respectively, one kilogram, 800 grams and five and one-half kilograms.

The exterior coating was dull. The interior in many parts is of a dark gray color, and in other parts quite light. The principal cause of the dark color is, doubtless, the larger amount of nickeliferous iron in that part. The specific gravity was 2.601.

Its composition was

Nickeliferous iron	15.21
Stony minerals	84.79

The nickeliferous iron consisted of

Iron	92.12
Nickel	6.20
Cobalt	.41

Copper and phosphorus not estimated.

The stony part, when treated with a mixture of hydrochloric and nitric acids, gave: insoluble part, 47.02; soluble part, 52.98. The former consisted of

Silica	52.61
Alumina	4 80
Ferrous oxide	13.21
Magnesia	27.31
Alkalies (soda, with traces of potash and lithia)	1.38
	99.31

and is essentially bronzite. The soluble portion gave:

Silica	38.01
Ferrous oxide	17.51
Magnesia	41.27
Alumina	.46
Sulphur	1.01

This is evidently olivine, with a small amount of sulphide of iron so disseminated through the stone that it is not easily separated by mechanical means. The meteorite then consists of nickeliferous iron, bronzite and olivine with small particles of anorthite and enstalite.

Literature—Amer. Jour. Sci., 3d Ser. x, p. 147; Min. and Min. Loc., p. 16; Smithson. Rep., 1885-'86, Part II, pp. 258, 262; Huntington, p. 94; Kerr, p. 314; Smith's Res., p. 478.

Present Possessors—Harvard, 211 grams; National Museum, 19.7; London, 29.4; Vienna, 285; Yale, 248; Bailey, 9.8; Baumhauer, 40; Gregory, 10.5.

20.

Randolph Meteorite.

Locality—Randolph county. *Analyst*—Shepard.

This was first described by Prof. Olmsted in 1822, in a descriptive catalogue of rocks and minerals collected by him, during his geological survey of North Carolina. It is there spoken of as occurring in the vicinity of a bed of argillaceous iron ore. It is distinctly foliated, the laminæ being thin and much interlaced. It weighed originally about two pounds. When etched it presents very fine, almost invisible, feathery lines much resembling hoar frost on a window-pane. Hardness equal to that of the best tempered steel. Specific gravity=7.618. The only metal detected, besides iron, was cobalt, which was present in traces only. A reddish brown powder, insoluble in *aqua regia*, was considered to be silicon.

Literature—Amer. Jour. Sci., 1st v, p. 262; 2d IV, p. 85; Jahresber., 1847-'48, p. 1311; Clark, p. 75; Min. and Min. Loc., p. 13; Kerr Appendix, p. 56; Buchner, p. 160.

Present Possessors—This is reported by Buchner as in collection of Amer. Geolog. Society and in London.

21.

Rockingham Meteorite.

Locality—Smith's Mt., Rockingham county. *Analysts*—Genth and Smith.

This was found in 1866 at Smith's Mountain, two miles north of Madison, in an old field grown up with pines, but cultivated ten or fifteen years previously. It fell probably in the interval. The original weight was eleven pounds. It is highly crystalline and on etching gives fine Widmanstätten figures, showing that it consists of probably three kinds of iron. It contains also schreibersite in short, very minute quadratic crystals, and, according to J. L. Smith, solid chloride of iron. Specific gravity, 7.78.

Iron		90.41	90.88
Nickel }		8.74	8.08
Cobalt }			.50
Copper		.11	.03
Insoluble	Iron	.27	
Phosphide	Nickel (Cobalt)	.33	
	Phosphorus	.14	.03
		100.00	99.46

Literature—Min. and Min. Loc., p. 15; Kerr, p. 313; Kerr Appendix, p. 56; Scient. Res., p. 526.

Present Possessors—N. C. State Museum; Jardin des Plantes, Paris; London, 77.3 grams; Vienna, 124; Göttingen, 54; Harvard, 821; Nat. Mus., 58.8; Gregory, 8; Bailey, 128.

22.

Rockingham Meteorite.

Locality—Rockingham county. *Analyst*—Venable.

This mass was reported to have fallen about the year 1846 on Deep Springs Farm, Rockingham county. Its fall caused much terror among the negroes on the place. It was dug out immediately after falling, being buried four or five feet under the surface. After lying about the house for many years, it was in the fall of 1889 presented to the State Museum.

The weight of the mass was 11.5 kilos. It measured 270 x 210 mm., having a varying thickness of 10 to 70 mm. It is coated

with a crust of several millimetres thickness. The surface is irregularly pitted with broad, shallow pits. On being polished it gave faintly the Widmanstätten figures. It belongs to the class of sweating meteorites.

Analysis:

Iron	87.01
Phosphorus	.04
Silica	.53
Chlorine	.39
Nickel	11.69
Cobalt	.79
	100.45

Literature—Amer. Jour. Sci., 3d Ser., 1890, p. 161; Mitchell Soc., VII, p. 29.
Present Possessor—N. C. State Museum.

23.

Rutherfordton Meteorite.

Locality—Rutherfordton, Rutherford Co. *Analysts*—Shepard, Rammelsberg.

This was analyzed by Shepard, who found Fe.=84.00, Si.=13.57, P.=1.31. He called it "ferrosilicine." A partial analysis made in Wöhler's laboratory gave Fe.=87.1, Si.=10.6, C.=0.4. Rammelsberg on examining it declared it to be nothing more than a piece of white pig iron of inferior quality.

This is placed among pseudo-meteorites in the Catalogue of the British Museum.

Literature—Amer. Jour. Sci., 2d XXXIV, p. 298; Kerr Appendix, p. 56; Clark, p. 67; J. prakt. chem., LXXXV, 87.
Present Possessor—Amherst (?)

24.

Rutherford Meteorite.

Locality—Ellenboro, Rutherford county. *Analyst*—Eakins.

This iron was found in the latter part of 1880, on a farm near Ellenboro, Rutherford county, N. C. Its nature remained unknown until February, 1890, when it was brought for examination to Mr. Stuart W. Cramer, of the U. S. Assay Office at Charlotte, N. C. It seems to have weighed about $2\frac{1}{5}$ kilos. In

shape it was roughly two globular ends with a connecting bar, the total length being about 150 mm. with end diameters 75 mm., and 50 mm. in the middle.

The iron is very tough and highly crystalline, the Widmanstätten figures showing distinctly on a polished, unetched face, and after etching they are unusually strong, Small, irregularly distributed patches of troilite are visible, and schreibersite also seems to be present. The analysis is as follows:

Fe	88.05
Ni	10.37
Co	.68
Cu	.04
P	.21
S	.08
Si	.02
	99.45

Literature—Amer. Jour. Sci., 1890, p. 395.

Present Possessors—Charlotte Assay Office and Geo. F. Kunz.

LIST OF WORKS REFERRED TO.

Some of these works are referred to from citations only. The limitation of library facilities prevented direct reference.

Amer. Jour. Sci.—American Journal of Science and Arts. Three Series.

Jahresbericht—Jahresbericht über die Fortschritte der, etc., Chemie, etc. Liebig and Kopp.

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Huntington—Catalogue of all Recorded Meteorites, 1887.

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Min. and Min. Loc.—The Minerals and Mineral Localities of North Carolina, 1885.

Scient. Researches—Mineralogy and Chemistry, Original Researches. J. L. Smith, 1873.

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Kerr Appendix—Appendix to Kerr's Geology.

Mitchell Soc.—Journal of Elisha Mitchell Scientific Society.

Partsch—Die Meteoriten in Hof-Mineralien-Kabinette zu Wien, 1843.

Buchner—Die Meteoriten in Sammlungen, 1863.

Gilb. Ann.—Gilbert's Annalen der Physik.

Chladni—Ueber Feuer-Meteore und über die mit denselben herabgefallenen Massen, 1819.

NO.	COUNTY.	ACCREDITED NAME.	DATE.	WEIGHT.	PAGE.
1	Alexander Co.	.....	secured 1875.....	56 grams.....	33
2	Ashe "	.....	.....	.....	34
3	Buncombe "	Asheville	found 1839.....	13630 grams.....	35
4	" "	Black Mountain..	" 1835.....	590 grams.....	36
5	" "	Hominy Creek....	" 1845.....	2000-3000 grams.....	37
6	Burke "	Linville.....	" 1842.....	442 grams.....	38
7	" "	Bridgewater.....	" 1888.....	13630 grams.....	39
8	Cabarrus "	.....	fell 1849.....	8860 grams.....	39
9	Caldwell "	.....	.....	5½ grams.....	40
10	Caswell "	.....	fell 1810.....	1360 grams.....	41
11	Davidson "	.....	found 1879.....	12720 grams.....	41
12	Guilford "	.....	described 1841..	1250 grams.....	42
13	Haywood "	.....	described 1854..	3.6 grams	43
14	" "	Ferguson.....	fell 1889.....	227 grams.....	43
16	Madison "	Jewel Hill.....	found 1856.....	18180 grams.....	44
17	" "	" "	" 1854.....	4000 grams.....	44
18	" "	Duel Hill	" 1873.....	11360 grams.....	45
19	Nash "	.....	fell 1874	(3) 1000, 800, & 5500 grams.	46
20	Randolph "	.....	described 1822..	900 grams.....	47
21	Rockingham "	Smith's Mountain	found 1866.....	5000 grams.....	48
22	" "	.....	fell 1846	11500 grams.....	48
23	Rutherford "	Rutherfordton.. ..	.....	.....	49
24	"	.....	found 1880.....	2500 grams.....	49

NEW AND IMPROVED METHODS OF ANALYSIS.

BY S. J. HINSDALE.

COLORIMETRIC METHOD FOR ESTIMATING THE MORPHINE STRENGTH OF LAUDANUM AND OTHER PREPARATIONS OF OPIUM.

Prepare an officinal tincture of opium with assayed opium. You will know the morphine strength of this tincture.

Make three dilutions of it with dilute alcohol, as follows:

One	3	parts	tincture	and	1	part	dilute alcohol.
One	2	"	"	"	2	"	"
One	1	"	"	"	3	"	"

Put 12 cc. of the tincture and of the dilutions in vials, and add to each 12 cc. dilute alcohol—cork well and keep them as standard dilutions of known strength. Label them Nos. 1, 2, 3 and 4. Let the dilute officinal tincture be No. 1. Dissolve 0.04 gram potassic ferrideyanide in 500 cc. water, and add to it fifteen drops liquor ferri chloridi. Call this *Ferrideyanide Mixture*. (*This must be freshly prepared*). Prepare it in a glass-stoppered bottle, with water perfectly free of iron.

Place four 50 cc. clean glass tumblers or wine-glasses on a white surface, and deliver *with a pipette* (about one-third filled) one drop of the dilutions in the glasses, commencing with No. 4 (the weakest), blowing out the pipette after each dropping. (The pipette should be about four inches long, and made of one-quarter inch tubing, and should deliver drops of the dilutions weighing *about* .016 gram or one-fourth grain. To test the pipette, see how many drops will balance a .200 gram weight. The reason for using so small a drop, and for diluting the tincture, is because a full drop of the undiluted tincture would develop too deep a blue color).

Now add to each glass about 5 cc. ferrideyanide mixture (it is convenient to use a homeopathic vial as a measure), and in

about one minute add 15 or 20 cc. water, and observe the shades of color. This observation must be made *within five minutes*, as the air and light will soon cause all to be uniformly blue.

By comparison with the shades of color produced by these standard dilutions, you can easily estimate the strength of any sample of laudanum with much accuracy. The sample must, of course, be diluted with an *equal part of dilute alcohol.* The presence of tannin interferes with this method, but opium does not contain tannin. Tannin is easily detected with a solution of a salt of iron. The ferridcyanide mixture *must be freshly prepared* and the glasses must be *clean and clear*, as the slightest bluish tinge interferes. Wash them with caustic soda and then with hydrochloric acid and rinse if they are soiled with Turnbull's Blue.

The ferridcyanide mixture is probably the best confirmatory test for morphine. If one drop of water containing .000001 gram of morphine is mixed on a white slab with one drop of the ferridcyanide mixture a blue color will be developed within *one* minute. With water alone the mixture will become of a bluish shade in about *ten* minutes, owing to the action of air and light.

P. S.—To estimate the strength of vinous or aqueous compounds of opium they must be brought to about the same specific gravity as the "standard dilutions" with alcohol, that the drops may be uniform in size.

COLORIMETRIC METHOD FOR ESTIMATING TANNIN IN BARKS, ETC.

Dissolve 0.04 gram potassic ferridcyanide in 500 cc. water, and add to it 1.5 cc. (about 22 drops) liquor ferri chloridi. Call this *Iron Mixture.*

Dissolve 0.04 gram "pure tannin (gallotannic acid) which has been dried at 212° F. in 500 cc. of water. Call this *Tannin Solution.*

Exhaust 0.8 gram oak bark with boiling water, and make it up to 500 cc. with cold water.

Place six two-ounce clear glass tumblers (or beaker glasses) on a white surface, and in one of them, *with a dropping pipette* (about four inches long and one-quarter inch wide) *about half filled*, put *five drops* of the infusion of bark, and in the others, *with the same pipette* (after rinsing), put 4, 5, 6, 7 and 8 drops of the "tannin solution." (The drops of the infusion and of the tannin solution must be uniform. The use of the same pipette, about half filled, *insures that*).

Now, add to each 5 cc. of "iron mixture," and in about one minute add to each tumbler about 20 cc. water, and *within three minutes* observe the shades of color. The number of drops of "tannin solution" used in the tumbler which corresponds in shade of color to the tumbler containing the infusion of bark *indicates the percentage of tannin in the bark; i. e.*, if it is the one in which seven drops were placed, the tannin strength of the bark is *seven per cent.*

It is best to observe the shades of color horizontally, rather than vertically, and to hold up the infusion tumbler, with the one which most nearly corresponds, opposite to a white wall, with your back to the light.

The above is written for *oak bark*, but the same process will answer for any substance containing less than ten per cent. of tannin.

For substances containing between about 10 and 20 per cent., it is best to dilute the infusion with an equal part of water and proceed as above, using *five drops* of the *dilute* infusion, and for the answer *double the result*. Thus, if the *diluted* infusion of tea required eight drops tannin solution to correspond, call the percentage *sixteen*.

For substances containing less than one, or one and a half per cent., exhaust 8 *grams* instead of 0.8 *gram*, and take *one-tenth* of the result for the answer. For substances containing more than twenty per cent., as galls, sumach, catechu, etc., you may dilute the infusion with two, three or more times its bulk with

water, and calculate as above (as with tea), or you may use 1, 2, 3 or 4 drops of the undiluted infusion in the first glass and make the calculation thus, *i. e.:* As the number of drops of infusion used is to the number of drops "tannin solution" used (to correspond), so is 5 to the answer—thus, suppose *two* drops infusion were used and the corresponding tumbler contained *fifteen* drops tannin solution—2 : 15 : : 5, answer 37.5 per cent.

The object in diluting the infusions is because the infusion glass may be of too deep a blue shade. It is better that it should just produce a *light blue.*

The tumblers must be perfectly clear and clean.

The "iron mixture," "tannin solution" and infusion must be freshly prepared and not exposed to the rays of the sun.

The water used must be free of iron and tannin.

The results are necessarily in terms for commercial gallotannic acid, and not for those of pure tannin, or of the particular tannin in the material assayed.

TEST FOR IRON.

A solution of neutral sulphite of soda containing a little pyrogallic acid has been proposed as a test for copper. A few drops of it mixed with a dilute solution of a salt of copper produces a red color similar to that which is developed by the addition of sulphocyanide of potash to a solution of a persalt of iron. The test is much more delicate for *iron,* as the following experiment will show:

Dissolve 0.7 gram ammonia ferrous sulphate (= 0.1 gram iron) in a liter of water; it will be 1 part in 10,000. To 10 cc. of this solution add water to make 100 cc.; this will be 1 to 100,000. Dilute some of this by adding four times its bulk of water; it will then be 1 in 500,000.

Make a saturated solution of sodium sulphite, and separately a solution of pyrogallic acid 0.5 gram in 50 cc. water. Put some of the iron solution in a wine-glass, add 4 drops of the

solution of sodium sulphite and afterward 2 drops of the pyrogallic solution and a purple color will be developed.

This test with distilled water alone develops a light pink shade, which, however, soon fades. This is due to the trace of free ammonia which it usually contains. Iron produces a purple tint. The test is so delicate that it will detect iron in 100 cc. of water, in which a bright cambric needle has been immersed for an hour.

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